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(54) PRODUCTION OF HARD COMPOSITE MATERIAL CONTAINING CUBIC BORON NITRIDE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a high-density composite material having excellent toughness, chipping resistance, strength and workability by subjecting a mixture composed of powders of specific compsns. to pressure sintering under and at a specific pressure and temp.

SOLUTION: The compsn. of the mixture is composed, by volumetric %, of 30 to 65% cubic boron nitride powder, 20 to 50% aluminum oxide powder, 0.1 to 10% metallic aluminum powder, 0.1 to 8% ≥ 1 kinds of compd. consisting of any of Li, Na and K and oxygen and silicon and 10 to 30% powder of ≥ 1 kinds of any of the carbide, nitride and boride of Si and periodic table group 4A, 5A, 6A elements. The mixture is subjected to pressure sintering under 100MPa pressure and a 1000 to 1500°C. As a result, the need for a coating stage requiring costly devices is eliminated and highly dense sintered compacts are produced relatively easily.

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TITLE: Manufacturing hard composite material containing cubic boron mixture - comprises **cubic boron nitride**, alumina, aluminium@, silicon, oxide of Gp=I element and carbide, boride or nitride of Gp=IVA, Gp=VA and Gp=VIA

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Basic Abstract Text - ABTX (1):

The mixture comprises (vol.%): **CBN** 30-65, **Al₂O₃** powder 20-50; **Al** powder 0.1-10; compound of one of Li, Na, K with O; Si 0.1-8; one of carbide, nitride, boride of Groups 4A, 5A, 6A 10-30. It is sintered at 1000-1500 deg. C at a pressure above 100 MPa.

Basic Abstract Text - ABTX (3):

ADVANTAGE - The hard composite material containing **CBN** is manufactured at relatively low temperature and low pressure.

Title - TIX (1):

Manufacturing hard composite material containing cubic boron mixture - comprises **cubic boron nitride**, alumina, aluminium@, silicon, oxide of Gp=I element and carbide, boride or nitride of Gp=IVA, Gp=VA and Gp=VIA

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(54) 【発明の名称】 立方晶窒化硼素含有硬質複合材料の製造方法

(57) 【要約】

【課題】 本来立方晶窒化硼素が準安定な状態となるような比較的低い圧力、温度の加圧焼結条件により、予め被覆処理等を行った原料を用いることなく、六方晶窒化硼素を含まない高緻密な立方晶窒化硼素含有硬質複合材料を優れた生産性で製造する方法を提供する。

【解決手段】 立方晶窒化硼素、酸化アルミニウム、金属アルミニウム、周期律表4A、5A、6A族元素及びSiの何れかの炭化物、窒化物、硼化物を1種以上、及びアルカリ金属と酸素と珪素からなる化合物1種以上からなる混合物を100MPa以上、1000~1500℃で加圧焼結する。

【特許請求の範囲】

【請求項1】 立方晶窒化硼素粉末30～65容量%、酸化アルミニウム粉末20～50容積%、金属アルミニウム粉末0.1～10容量%、Li、Na、Kの何れかと酸素と珪素からなる化合物1種以上を0.1～8容量%、Si及び周期律表4A、5A、6A族元素の炭化物、窒化物、硼化物の何れか1種以上の粉末を10～30容量%含む混合物を、圧力100MPa以上、温度1000～1500℃で加圧焼結することを特徴とする立方晶窒化硼素含有硬質複合材料の製造方法。

【請求項2】 Li、Na、Kの何れかと酸素と珪素からなる化合物が、メタ珪酸リチウム、メタ珪酸ナトリウム、メタ珪酸カリウムの何れかの水和物又は無水塩であることを特徴とする請求項1記載の立方晶窒化硼素含有硬質複合材料の製造方法。

【請求項3】 圧力500～2000MPa、温度1100～1250℃で加圧焼結することを特徴とする請求項1又は2記載の立方晶窒化硼素含有硬質複合材料の製造方法。

【発明の詳細な説明】

【0001】

【発明が属する技術分野】本発明は耐摩耗部材や切削用工具部材などに適用できる高硬度を有する立方晶窒化硼素含有無機系複合材料の製造方法に関する。

【0002】

【従来の技術】立方晶窒化硼素（以下cBNという）は、ダイヤモンドに次ぐ高い硬度であり、動的及び静的耐摩耗材として広く使用されているアルミナ等のセラミックス部材に比較して極めて高い耐摩耗性を示す。さらにcBNは高い熱伝導度を有すると共に常温から高温に至るまで鉄族金属との濡れ性がダイヤモンドに比較して著しく小さいことからその焼結体は、鋳鉄、高硬度鋼、及び従来の部材では切削が困難とされている耐熱合金等に対する切削工具用部材として極めて適している。

【0003】一般に、cBN焼結体を得るためには5000MPa以上の超高压で焼結されている。これはcBNが高圧安定型の変態であるため、低圧安定型の六方晶窒化硼素（以下、hBNという）への相転移を防ぐためである。しかしながら超高压焼結は他の焼結方法に比べ生産コストが著しく高く、必ずしも生産性が優れているとは云えない為、超高压焼結によって製造された硬質材料は低圧力で製造された硬質セラミックス、サーメット及び超硬合金等に比較すると価格が数十倍以上と極めて高価なものになっている。

【0004】この為、cBNにセラミックス、サーメット、超硬合金等の比較的高融点の無機物質を加えて複合化することによって焼結体製造時の圧力を低下させることが行われてきた。このようなcBN含有無機複合焼結体は、本来cBNが準安定となる圧力、例えば2000MPa以下、及び温度、例えば1500℃未満の条件下

であってもcBNの相転移速度を著しく低減することができ、実質的にはhBNを生成することなく、ある程度緻密なcBN含有無機複合焼結体を作製することが可能であることが報告されている（平成2年特許公開第302371号）。しかしこのような製造方法では、複合焼結体に占めるcBNの存在割合が多くなるにつれhBNへの転移を抑えて高緻密な焼結体を得るのが困難になり、一方cBNの存在割合が少なくなるにつれ焼結体に反映されるcBN本来の性状が急激に失われる傾向がある。更に、該製法による複合焼結体ではcBN含有量の多少に拘わらず、cBNと該無機物質との強固な結合が見られないので、機械的な性状面に於いては必ずしも優れているとは云い難く、複合化による性状強化が十分発揮され難かった。

【0005】一方、cBN粉末を主に高融点の無機物質で予め被覆し、これを原料として焼結したものは、前記のようなcBNの準安定な圧力温度域に於いても比較的高緻密な焼結体が得られることが報告されている。（平成7年特許公開第172923号、平成7年特許公開第53270号）この場合、焼結圧力を低下させることによってもたらされる生産コストの低下が期待されるものの、予め個々のcBN粒子全てに満遍なく被覆する必要があり、また一般に反応活性に乏しい高融点化合物の被覆はCVD法やプラズマ法によって処理しなければ十分な密着性を有する被覆物を製造することができず、このような煩雑で制約が多い被覆工程が加わる上に高価な製造装置を必要とする為、生産性の著しい向上は期待できない。

【0006】

【発明が解決しようとする課題】本発明は、被覆処理を行ったcBN粉末を用いず、本来cBNが準安定状態となるような比較的低い圧力、温度の加圧焼結条件により、機械的性状が脆弱なhBNを生成させることなく、かつ分散相としてのcBNが有する優れた耐摩耗特性及び基材相としての高融点無機物質が有する強度特性等も極力損なうことなく両相が強固に結合したcBN含有無機系硬質複合材料をより低コストで製造することを目的とする。

【0007】

【課題を解決するための手段】本発明者らは前記の目的に鑑み、種々研究を行った結果、予め高融点無機化合物で被覆を施したcBN粉末を用いることなく、cBN粉末と酸化アルミニウム粉末、金属アルミニウム粉末、周期律表4A、5A、6A族の元素及びSiの何れかの炭化物、窒化物、硼化物の何れか1種またはこれら2種以上を組み合わせた高融点硬質無機系粉末に低融点化合物であるLi、Na、Kの何れかと酸素と珪素からなる化合物1種以上を混合したものを、本来cBNが準安定な状態となるような焼結条件で加圧焼結することにより、cBNがhBNに相転移することなく、cBN分散粒子

が高融点硬質無機系基材に強固に結合した高緻密な複合焼結体が容易にかつ比較的安価に得られることを見出し、本発明を完成するに至った。

【0008】即ち、本発明は、立方晶窒化硼素粉末30～65容量%、酸化アルミニウム粉末20～50容積%、金属アルミニウム粉末0.1～10容量%、Li、Na、Kの何れかと酸素と硼素からなる化合物1種以上を0.1～8容量%、Si及び周期律表4A、5A、6A族元素の炭化物、窒化物、硼化物の何れか1種以上の粉末を10～30容量%含む混合物を、圧力100MPa以上、温度1000～1500℃で加圧焼結すること

を特徴とする立方晶窒化硼素含有硬質複合材料の製造方法である。

【0009】また、本発明は、前記Li、Na、Kの何れかと酸素と珪素からなる化合物が、メタ珪酸リチウム、メタ珪酸ナトリウム、メタ珪酸カリウムの何れかの水和物又は無水塩であることを特徴とする立方晶窒化硼素含有硬質複合材料の製造方法である。

【0010】また、本発明は、立方晶窒化硼素粉末30～65容量%、酸化アルミニウム粉末20～50容積%、金属アルミニウム粉末0.1～10容量%、Li、Na、Kの何れかと酸素と硼素からなる化合物1種以上、又は該Li、Na、Kの何れかと酸素と珪素からなる化合物が、メタ珪酸リチウム、メタ珪酸ナトリウム、メタ珪酸カリウムの何れかの水和物又は無水塩であるもの1種以上を0.1～8容量%、Si及び周期律表4A、5A、6A族元素の炭化物、窒化物、硼化物の何れか1種以上の粉末を10～30容量%含む混合物を、圧力が500～2000MPa、温度が1100～1250℃で加圧焼結すること

を特徴とする立方晶窒化硼素含有硬質複合材料の製造方法である。

【0011】

【発明の実施の形態】本発明の方法によって製造される立方晶窒化硼素含有硬質複合材料の形態としては、cBNを除く高融点硬質無機物質を主体とする基材相中にcBN粒子が分散相として存在する緻密質の焼結体である。該硬質複合材料を得るための原料としては、cBN粉末、酸化アルミニウム粉末、金属アルミニウム粉末及びSi及び周期律表4A、5A、6A族の元素の炭化物、窒化物、硼化物より選択された1種以上の金属化合物若しくは固溶体からなる粉末、更にLi、Na、Kの酸素と珪素を含む化合物何れか1種以上の粉末を混合したものを用いる。以下本発明で用いる各原料について詳しく説明する。

【0012】原料に用いるcBN粉末としては、粒径が100μm未満の粉末が望ましい。粒径が100μm以上では強度の低下が顕著となるので好ましくない。このcBN粉末の原料混合物中に於ける配合量は30～65容量%、望ましくは45～65容量%とする。30容量%未満ではcBN固有の硬度を十分活かした優れた耐摩

耗性を有する複合材料が得られず、65容量%を越えると、複合材料中でのcBN量が過多となり、cBN以外の成分を主体とする連続したセラミックス基材相が形成され難くなり、その結果、高緻密で高強度の材料を得ることができなくなるので好ましくない。

【0013】酸化アルミニウム粉末としては比較的純度の高い市販品であれば何れのものでも用いることができる。原料混合物中に於ける酸化アルミニウム配合量は20～50容量%、望ましくは25～40容量%とする。20容量%未満ではcBNと酸化アルミニウム以外の物質の存在割合が多くなって焼結を行う上でより高温、高圧を必要とするので好ましくなく、また50容量%を越えると、cBNや酸化アルミニウム以外の成分の割合が少なくなり得られた複合材料の耐摩耗特性や強度及び耐熱性や熱衝撃抵抗が低下するので好ましくない。

【0014】Siの炭化物、窒化物、硼化物の粉末としては市販のSiC粉末、Si₃N₄粉末、SiB₄粉末を用いることができ、このような金属珪素化合物に於いてはそれ自体の高緻密焼結の際通常使用されている公知の焼結助剤が含まれたものを用いても良い。また、原料に用いる周期律表4A、5A、6A族元素の炭化物、窒化物、硼化物の粉末としてはTiC、ZrC、HfC、VC、NbC、TaC、Cr₃C₂、Mo₂C、WC、TiN、ZrN、HfN、VN、NbN、Ta₃N₂、Mo₂N、WN、TiB₂、ZrB₂、HfB₂、VB₂、NbB₂、TaB₂、Cr₃B₄、MoB₂、WB₂等の各粉末、又はこのうち2種以上からなる混合粉末、或いはTiC、ZrC、HfC、VC、NbC、TaC、Cr₃C₂、Mo₂C、WC、TiN、ZrN、HfN、VN、NbN、Ta₃N₂、Mo₂N、WN、TiB₂、ZrB₂、HfB₂、VB₂、NbB₂、TaB₂、Cr₃B₄、MoB₂、WB₂の何れか2種以上からなる固溶体粉末を用いる。このSi及び周期律表4A、5A、6A族元素の炭化物、窒化物、硼化物の何れか1種以上の粉末の原料混合物中での配合量は10～30容量%、より望ましくは15～25容量%とする。10容量%未満では前記成分に概ね共通する高温特性、即ち高温高硬度、高温高強度等が十分反映された複合材料が得難くなり、30容量%を越えると難焼結成分の含有比率が大となって緻密化がかなり進み難くなるので好ましくない。

【0015】金属アルミニウム粉末としては、市販の金属アルミニウムの高純度粉末を用いることができ、その原料混合物中の配合量は0.1～10容量%とする。金属アルミニウム粉末の配合量が0.1重量%未満では焼結体の密度や強度の低下となり、10容量%を越えると溶融Alが基材相中に析出して複合材料の機械的性質が著しく低下することがあるので好ましくない。

【0016】また、原料に用いるLi、Na、Kの酸素と珪素を含む化合物としては、例えばLi₂SiO₃、Li₄SiO₄、Li₂SiO₃・H₂O、Li₆Si₂O₇、N

$a_2\text{SiO}_3$ 、 Na_4SiO_4 、 $\text{Na}_2\text{Si}_2\text{O}_5$ 、 $\text{Na}_2\text{Si}_4\text{O}_9$ 、 $\text{Na}_2\text{O} \cdot x\text{SiO}_2 \cdot n\text{H}_2\text{O}$ (但し、 $x=2\sim 4$ 、 $n\geq 1$)、 K_2SiO_3 等を挙げることができ、これらのうちから適宜選択されたものを用いる。その原料混合物中の配合量は0.1～8容量%とする。0.1容量%未満では緻密化が不足したり、cBN粒と基材相との結合状態が低下し、例えば強度低下や脱粒等が生じ易くなるので好ましくない。また8容量%を越えると焼結体中に存在するLi、Na、Kの何れか1種以上を含む低融点物質の割合が増大し、硬度を始め強度や靱性などの機械的性質が低下することがあるので好ましくない。尚、前記外のアルカリ金属、即ちRb、Csについてもその酸素と珪素を含む化合物を同様に用いることができる。

【0017】これらの原料を用いて立方晶窒化硼素含有硬質複合材料を得るまでの製造工程の詳細を以下に示す。各原料成分は前記所定の配合量になるように秤量、混合し、原料混合物を作製する。原料の混合は、公知の湿式混合、例えばエチルアルコール或いはイソプロピルアルコール等の有機溶媒を用いてボールミル中で混合することに対応できるが、本発明では有機溶媒の代わりに水などの無機系溶媒を用いることもできる。次いでこの原料混合物を必要に応じ減圧乾燥や噴霧乾燥等で適宜乾燥し、乾燥原料混合物をプレス成形等で所望の形状に成形した成形物を加圧焼結するか、或いは未成形の原料混合物を所望形状の加圧用セルに入れて加圧焼結する。

【0018】加圧焼結は、圧力100MPa以上、温度1000～1500℃で行う。例えばcBNが単独で安定に存在する領域で加圧焼結するのであれば圧力500～6000MPa、温度1400～1500℃が一般的な条件となるが、cBNが単独では準安定に存在すると考えられる比較的低い圧力、温度領域では製造コストの著しい低減化が図れ、本発明ではこのような加圧温度条件に於いても複合材料中でcBNが脆弱相であるhBNへ相転移することを十分抑えることができる為、cBNが単独では準安定に存在すると考えられる圧力及び温度条件で加圧焼結することが特に推奨される。このような条件として好ましくは、圧力500～2000MPa、温度1100～1250℃で行うのが良い。一方、温度1000℃未満では焼結が十分進展し難く緻密化が不十分になり易く、逆に温度が1500℃を越える場合や圧力が100MPa未満では、cBNが非安定とされている領域となるため、脆弱相であるhBNへの相転移が生じることがあるので好ましくない。また加圧焼結の保持時間は圧力と温度に依存するが、概ね5～30分程度で対応できる。尚、焼結時の雰囲気についてはcBNや基材相の非酸化成分の酸化を直接引き起こすような高い酸化性雰囲気でない限りは特に限定されず、例えば空気中でも可能であるが、より望ましくは真空中やアルゴンガス等の不活性ガス中で焼結すると良い。

【0019】このような加圧焼結に用いる装置としては圧力100MPa以上、温度1000℃以上を発生できる装置であれば何れの装置でも良い。そのような装置の一例としては、ホットプレス装置、HIP装置、ピストンシリンダー型、フラットベルト型、ガードル型、ブリッジマン型、キュービックアンビル型等の高圧、又は超高圧加熱装置を挙げることができる。このような条件、装置にて加圧焼結を行うことにより、hBNを含まないcBN粒子が、酸化アルミニウムと微量のムライトやその固溶体及び、Si及び周期律表4A、5A、6A族元素の炭化物、窒化物、硼化物の何れか1種以上の成分を主体とし、これにアルカリ金属であるLi、Na、Kの酸素及び/又は珪素を含む物質の何れか1種以上が加わったものから成る連続した基材中に分散された高緻密な立方晶窒化硼素含有硬質複合材料を製造することができる。

【0020】

【作用と効果】本発明に於けるLi、Na、Kのようなアルカリ金属の酸素と珪素を含有する化合物は、加圧焼結の前又は初期段階においてcBN粒子、及び他の基材相構成成分とそれぞれ反応若しくは固溶し、cBN粒子と基材相との間の反応活性を増大させ、その結果、複合焼結体中で分散相と基材相との間に比較的強固で化学的な結合状態が形成されると考えられ、その結果焼結体中でcBN粒子は基材相に強く保持されて脱粒が起こり難くなる他、焼結体の強度向上にも繋がる。更に温昇過程で、アルカリ金属の酸素と珪素を含む化合物の一部の珪酸成分が分離し、該珪酸成分が基材相中で、特に酸化アルミニウム表面部と反応し酸化アルミニウムの粒界に高融点化合物であるムライトやその固溶体を生成する。このムライトは、基材相を構成する他の高融点硬質無機物質に比べ熱膨張率が低く、基材相の熱衝撃抵抗を向上させる。

【0021】また、アルカリ金属の珪酸塩は、水系溶媒中での分散効果が優れる為、本発明では原料調整段階に於いて有機系以外の溶媒、即ち、無機系溶媒である水を用いることもできる。従って、取り扱いがより簡易になり、コストの低減化にも結びつく他、有機系溶媒を用いたときに屢々生じる加熱後の残存炭素不純物による焼結体性状の劣化を回避することができる。

【0022】また更に、本発明によれば、cBN粉末は例えば高融点無機化合物で予め被覆しなくとも、cBNの準安定領域といわれている圧力、温度でhBNを生成することなく十分緻密な高硬度の複合焼結体を製造することができる。この理由についてはcBN粒の界面付近に比較的低温の焼結の前段階から低融点のアルカリ金属と珪素とを含む化合物がcBN本体表面部と反応し、この反応生成物がcBN表面に形成されることによりhBNへの相転移を抑制するものと考えられる。このように本法は従来方法よりも特に焼結圧力を低くすることがで

き、かつ高価な装置を要す被覆工程を不要とし、しかも比較的容易に高緻密な焼結体を製造することができるため生産性の大幅な向上と生産コストの大幅な低減化が可能となる。本発明は、高硬度、高耐摩耗性のみならず、優れた靱性、耐欠損性、強度、及び加工性を備えた高緻密な複合材料を得ることができる為、耐摩耗用途や金属切削用途の部材製造用としても極めて適した製造方法である。

【0023】

【実施例】以下、実施例及び比較例によりこの発明を具体的に詳しく説明する。

【実施例1】 平均粒径0.5 μ mの酸化アルミニウム粉末、平均粒径3.0 μ mの金属アルミニウム粉末、平均粒径1 μ mの窒化チタン粉末（純度99.5%）、平均粒径1 μ mの硼化タンタル粉末（純度99.5%）、平均粒径約1 μ mの炭化タングステン粉末（純度99.5%）、平均粒径約1 μ mの窒化珪素粉末（純度99.*

*5%)、平均粒径約3 μ mのcBN粉末、平均粒径約5 μ mのメタ珪酸リチウム粉末、平均粒径約1.0 μ mのメタ珪酸ナトリウム粉末、粘液状のメタ珪酸ナトリウム水和物、平均粒径約5 μ mのメタ珪酸カリウム粉末を、表1に記した7種類の原料配合割合となるようそれぞれアルミナ製ボットに入れアルミナボールで72時間水を用いて湿式混合後、混合物を噴霧乾燥して顆粒を作成し、これをプレス成形にて直径約5cm厚さ約2cmの円柱形状に成形した。この成形物をピストンシリンダー型高压焼結炉を用いて空气中、1000MPa、1250℃で15分間加压焼結した。得られた焼結体（本発明品1～7）の成分を粉末X線解析により分析したが、何れもhBNは検出されず、各原料組成に概ね対応した高融点物質とcBNを主体とする成分及び微量のムライトが検出された。

【0024】

【表1】

本発明品	原料配合量		焼結体機械的性質
	原料名	容量%	
1	cBN	37.7	相対密度 99.9% ビッカース硬度 2900 曲げ強度 145MPa
	Al ₂ O ₃	32.5	
	Al	8.2	
	TiN	25.2	
	Li ₂ SiO ₃	1.4	
2	cBN	52.7	相対密度 99.9% ビッカース硬度 3000 曲げ強度 125MPa
	Al ₂ O ₃	21.8	
	Al	2.0	
	TiN	15.5	
	Na ₂ SiO ₃	8.0	
3	cBN	45.4	相対密度 99.9% ビッカース硬度 2800 曲げ強度 145MPa
	Al ₂ O ₃	22.4	
	Al	2.1	
	TiN	30.0	
	K ₂ SiO ₃	0.1	
4	cBN	30.0	相対密度 99.9% ビッカース硬度 2650 曲げ強度 130MPa
	Al ₂ O ₃	50.0	
	Al	3.2	
	TaB ₅	15.3	
	Na ₂ SiO ₃ ·2H ₂ O	1.5	
5	cBN	50.0	相対密度 99.9% ビッカース硬度 2950 曲げ強度 120MPa
	Al ₂ O ₃	20.0	
	Al	10.0	
	TiN	19.0	
	Na ₂ SiO ₃	1.0	
6	cBN	46.3	相対密度 99.9% ビッカース硬度 2750 曲げ強度 125MPa
	Al ₂ O ₃	41.6	
	Al	0.1	
	WC	11.9	
	Na ₂ SiO ₃	0.1	
7	cBN	65.0	相対密度 99.9% ビッカース硬度 2950 曲げ強度 140MPa
	Al ₂ O ₃	20.0	
	Al	2.5	
	Si ₃ N ₄	10.0	
	Na ₂ SiO ₃	2.5	

【0025】また、この焼結体の機械的性質を以下の如く測定し、測定値を表1に記す。尚、相対密度について※50 ※はJIS-C2141に準じた方法で測定した嵩比重と真比重から算出し、ビッカース硬度については圧子荷重

を5kgとして測定し、曲げ強度についてはJIS-R1601に準拠した三点曲げ強度(室温)を測定した。
 【0026】[実施例2] 平均粒径0.5 μ mの酸化アルミニウム粉末26容量%、平均粒径3.0 μ mの金属アルミニウム粉末2.5容量%、平均粒径約1 μ mの窒化チタン粉末(純度99.5%)20.1容量%、平均粒径約8 μ mのメタ珪酸ナトリウム粉末1.1容量%、平均粒径約3 μ mのcBN粉末50.3容量%を原料とし、前記実施例1と同様の方法にて直径約5cm厚*

*さ約2cmの円柱形状の未焼成成形物を作製した。これをピストンシリンダー型高圧焼結炉により表2に記す条件で加圧焼結するか(本発明品8~13)、又は、該成形物をSUS310製容器中に脱気封入し、Arガスを圧力媒体とした熱間等方加圧装置(HIP)にて表2に記す条件で加圧焼結を行い(本発明品14)、焼結体を作製した。

【0027】

【表2】

本発明品	加圧焼結条件			焼結体機械的性質
	温度(℃)	圧力(MPa)	焼結装置	
8	1000	1000	ピストンシリンダー型高圧焼結炉	相対密度 99.9% ビッカース硬度 2750 曲げ強度 135MPa
9	1100	1000	ピストンシリンダー型高圧焼結炉	相対密度 99.9% ビッカース硬度 2750 曲げ強度 136MPa
10	1200	1000	ピストンシリンダー型高圧焼結炉	相対密度 99.9% ビッカース硬度 3050 曲げ強度 140MPa
11	1500	1000	ピストンシリンダー型高圧焼結炉	相対密度 99.9% ビッカース硬度 3000 曲げ強度 135MPa
12	1250	2000	ピストンシリンダー型高圧焼結炉	相対密度 99.9% ビッカース硬度 3100 曲げ強度 140MPa
13	1250	500	ピストンシリンダー型高圧焼結炉	相対密度 99.9% ビッカース硬度 2900 曲げ強度 130MPa
14	1250	100	HIP	相対密度 99.7% ビッカース硬度 2700 曲げ強度 125MPa

【0028】得られた焼結体(本発明品8~14)の成分を粉末X線解析により分析したが、何れもhBNは検出されず、cBN、Al₂O₃、及びTiNを主体とする成分と微量のムライトが検出された。また、この焼結体の機械的性質を実施例1と同様の方法で測定した結果を表2に記す。

【0029】[比較例1] 平均粒径0.5 μ mの酸化アルミニウム粉末33.9容量%、平均粒径3.0 μ mの金属アルミニウム粉末3.2容量%、平均粒径約1 μ mの窒化チタン粉末(純度99.5%)25.2容量%、平均粒径約3 μ mのcBN粉末37.7容量%を原料とし、実施例1と同様の方法及び加圧焼結の条件にて直径約5cm厚さ約2cmの円柱形状の焼結体を作製した。該焼結体の機械的性質を実施例1と同様の方法で測定した結果、相対密度99.3%、ビッカース硬度2750、曲げ強度100MPaとなり曲げ強度がかなり低※

※いものとなった。

【0030】[比較例2] 平均粒径0.5 μ mの酸化アルミニウム粉末33.9容量%、平均粒径3.0 μ mの金属アルミニウム粉末3.2容量%、平均粒径約1 μ mの窒化チタン粉末(純度99.5%)25.2容量%、平均粒径約3 μ mのcBN粉末37.7容量%を原料とし、前記実施例1と同様の方法にて直径約5cm厚さ約2cmの円柱形状の未焼成成形物を作製した。これをSUS310製容器中に脱気封入にしてArガスを圧力媒体とした熱間等方加圧装置(HIP)にて圧力100MPa、温度1250℃で60分加圧焼結した。得られた焼結体の成分を粉末X線解析により分析した結果、hBNが検出され、また該焼結体の機械的性質を実施例1と同様の方法で測定した結果、相対密度92%、ビッカース硬度2300、曲げ強度105MPaとなり機械的性状が著しく低いものとなった。

rejection]

[Date of requesting appeal against examiner's
decision of rejection]

[Date of extinction of right]

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CLAIMS

[Claim(s)]

[Claim 1] 30 to cubic boron nitride powder 65 capacity %, 20 to aluminum-oxide-dust 50 volume %, One or more sorts of compounds which become any of 0.1 to metal aluminium-powder 10 capacity %, and Li, Na and K to be from oxygen and silicon 0.1 to 8 capacity %, The manufacture approach of the cubic boron nitride content hard composite material characterized by carrying out pressure sintering of the mixture 10-30 capacity % Containing any one or more sorts of powder of the carbide of Si and the periodic tables 4A and 5A, and 6A group element, a nitride, and boride at the pressure of 100 or more MPas, and the temperature of 1000-1500 degrees C.

[Claim 2] The manufacture approach of a cubic boron nitride content hard composite material according to claim 1 that the compound which becomes any of Li, Na, and K to be from oxygen and silicon is characterized by being which hydrate or anhydrous salt of a meta-silicic acid lithium, specific metasilicate, and a meta-potassium silicate.

[Claim 3] A pressure 500 - 2000MPa, the manufacture approach of a cubic boron nitride content hard composite material according to claim 1 or 2 characterized by carrying out pressure sintering at the temperature of 1100-1250 degrees C.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] This invention relates to the manufacture approach of cubic boron nitride content inorganic system composite material of having a high degree of hardness applicable to an antifriction member, the tool member for cutting, etc.

[0002]

[Description of the Prior Art] Cubic boron nitride (it is called Following cBN) is a high degree of hardness which ranks second to a diamond, and shows very high abrasion resistance as compared with ceramic members, such as dynamic and an alumina currently widely used as static antifriction material. Furthermore, since wettability with an iron-group metal is remarkably small as compared with a diamond until it results [from ordinary temperature] in an elevated temperature, while having thermal conductivity with high cBN, the sintered compact is extremely suitable as a member for cutting tools to cast iron, high degree-of-hardness steel, the heat-resistant alloy with which cutting is made difficult in the conventional member.

[0003] Generally, in order to obtain a cBN sintered compact, it is sintered with the extra-high voltage of 5000 or more MPas. Since cBN is the transformation of a high-pressure stability mold, this is for preventing the phase transition to the hexagonal boron nitride (henceforth hBN) of a low voltage stability mold. However, extra-high voltage sintering has a remarkably high production cost compared with other sintering approaches, and since it cannot necessarily say that productivity is excellent, if the hard material manufactured by extra-high voltage sintering is compared with hard ceramics, a cermet, cemented carbide, etc. which were manufactured under the low voltage force, it is what has very as expensive a price as dozens or more times.

[0004] For this reason, reducing the pressure at the time of sintered compact manufacture has been performed by the ceramics, a cermet, cemented carbide, etc. adding high-melting mineral matter comparatively, and compound-izing to cBN. It is reported that it is possible to produce a to some extent precise cBN content inorganic compound sintered compact, without generating hBN substantially, since the phase transition rate of cBN can be remarkably reduced even if such a cBN content inorganic compound sintered compact is the bottom of the less than pressure which becomes metastable [cBN] originally, for example, 2000 MPas, and temperature, for example, a less than 1500-degree C condition, (the Heisei 2 patent public presentation No. 302371). however -- as the abundance of cBN occupied to a compound sintered compact increases by such manufacture approach -- the transition to hBN -- stopping -- quantity -- it becomes difficult to obtain a precise sintered compact and, on the other hand, the abundance of cBN decreases -- be alike and hang -- there is an inclination for the description of cBN original reflected in a sintered compact to be lost rapidly. furthermore -- since firm association with cBN and this mineral matter is not seen irrespective of some of cBN contents in the compound sintered compact by this process -- mechanical description -- the description are hard to say that it not necessarily excels in a field, and according to compound-izing -- strengthening was sufficiently hard to be demonstrated.

[0005] even if it sets what mainly covered cBN powder with high-melting mineral matter beforehand, and sintered this as a raw material on the other hand in the metastable above pressure temperature regions of cBN -- comparatively -- quantity -- it is reported that a precise sintered compact is obtained. In this case (The Heisei 7 patent public presentation No. 172923, the Heisei 7 patent public presentation No. 53270) Although

the fall of the production cost brought about by reducing a sintering pressure is expected It is necessary to cover uniformly to each cBN particles of all beforehand and, and, generally covering of a high-melting compound lacking in labile cannot manufacture the coating which has sufficient adhesion if it processes neither by the CVD method nor the plasma method. It is complicated, and since a covering process with much constraint is added and also [such] an expensive manufacturing installation is needed, the remarkable improvement in productivity is not expectable.

[0006]

[Problem(s) to be Solved by the Invention] this invention is mechanical by the pressure-sintering conditions of a comparatively low pressure from which cBN will originally be in a metastable state, without using the cBN powder which performed covering processing, and temperature -- a bi-phase aims at manufacturing more the cBN content inorganic system hard composite material combined firmly by low cost, without [without it makes hBN with brittle description generate, and] spoiling the strength property which the high-melting mineral matter as the outstanding antiwear characteristic which cBN as a dispersed phase has, and a base material phase has as much as possible.

[0007]

[Means for Solving the Problem] this invention persons without using the cBN powder which covered with the high-melting inorganic compound beforehand, as a result of studying many things in view of the aforementioned purpose cBN powder, aluminum oxide dust, a metal aluminium powder, periodic table 4A, Li which is a low-melt point point compound at the high-melting hard inorganic system powder which combined any one sort of the element of 5A and 6A group and which carbide of Si, a nitride, and the boride, or these two sorts or more, By carrying out pressure sintering of what mixed Na and one or more sorts of compounds which become any of K to be from oxygen and silicon on sintering conditions from which cBN will originally be in a metastable condition the quantity which the cBN particulate material combined with the high-melting hard inorganic system base material firmly, without cBN carrying out phase transition to hBN -- it finds out that a precise compound sintered compact is obtained easily and comparatively cheaply, and came to complete this invention.

[0008] This invention Namely, 30 to cubic boron nitride powder 65 capacity %, 20 to aluminum-oxide-dust 50 volume %, One or more sorts of compounds which become any of 0.1 to metal aluminium-powder 10 capacity %, and Li, Na and K to be from oxygen and boron 0.1 to 8 capacity %, 10-30 capacity % It is the manufacture approach of the cubic boron nitride content hard composite material characterized by carrying out pressure sintering of the included mixture at the pressure of 100 or more MPas, and the temperature of 1000-1500 degrees C about any one or more sorts of powder of the carbide of Si and the periodic tables 4A and 5A, and 6A group element, a nitride, and boride.

[0009] Moreover, this invention is the manufacture approach of cubic boron nitride content hard composite material that the compound which becomes said any of Li, Na, and K to be from oxygen and silicon is characterized by being which hydrate or anhydrous salt of a meta-silicic acid lithium, specific metasilicate, and a meta-potassium silicate.

[0010] This invention Moreover, 30 to cubic boron nitride powder 65 capacity %, 20 to aluminum-oxide-dust 50 volume %, 0.1 to metal aluminium-powder 10 capacity %, one or more sorts of compounds which become any of Li, Na, and K to be from oxygen and boron, The compound which becomes any of this Li, and Na and K to be from oxygen and silicon Or a meta-silicic acid lithium, One or more sorts of things which are which hydrate or anhydrous salt of specific metasilicate and a meta-potassium silicate 0.1 to 8 capacity %, 10-30 capacity % Any one or more sorts of powder of the carbide of Si and the periodic tables 4A and 5A, and 6A group element, a nitride, and boride the included mixture It is the manufacture approach of the cubic boron nitride content hard composite material characterized by for a pressure carrying out by 500 - 2000MPa, and temperature carrying out pressure sintering at 1100-1250 degrees C.

[0011]

[Embodiment of the Invention] It is the sintered compact of the substantia compacta with which a cBN particle exists as a dispersed phase in the base material phase which makes a subject the high-melting hard mineral matter except cBN as a gestalt of the cubic boron nitride content hard composite material manufactured by the approach of this invention. the powder which consists of one or more sorts of the metallic compounds or the solid solutions which were chosen from the carbide of the element of cBN

powder, aluminum oxide dust, a metal aluminium powder, Si and the periodic tables 4A and 5A, and 6A group, a nitride, and boride as a raw material for obtaining this hard composite material, and the compound which contains the oxygen and silicon of Li, Na, and K further -- what mixed any or one or more sorts of powder is used. Each raw material used by this invention below is explained in detail.

[0012] As cBN powder used for a raw material, less than 100-micrometer powder has a desirable particle size. Since it becomes remarkable in 100 micrometers or more strong falling particle size, it is not desirable. the loadings in the raw material mixture of this cBN powder -- 30 to 65 capacity % -- it considers as 45 to 65 capacity % desirably. under by 30 capacity %, if the composite material which has the outstanding abrasion resistance which was able to harness the degree of hardness of a cBN proper enough is not obtained but 65 capacity % is exceeded, the amount of cBN(s) in the inside of composite material will become excessive, and the continuous ceramic base material phase which makes components other than cBN a subject will form -- having -- being hard -- consequently -- high -- it is precise, and since it becomes impossible that it is hard to obtain the ingredient of high intensity, it is not desirable.

[0013] Anything can be used if it is a commercial item with comparatively high purity as aluminum oxide dust. the aluminum-oxide loadings in raw material mixture -- 20 to 50 capacity % -- it considers as 25 to 40 capacity % desirably. Since an elevated temperature and high pressure are needed more when sintering by the abundance of matter other than cBN and an aluminum oxide increasing, it is not desirable, and if 50 capacity % is exceeded, since the antiwear characteristic of composite material, the reinforcement and thermal resistance whose rate of components other than cBN or an aluminum oxide might decrease, and thermal shock resistance will fall, it is not desirable at under 20 capacity %.

[0014] As powder of the carbide of Si, a nitride, and boride, commercial SiC powder, Si₃N₄ powder, and SiB₄ powder can be used, and that in which the well-known sintering acid usually used in such a metal silicon compound in the case of high precise sintering of itself was contained may be used. Moreover, the carbide of the periodic tables 4A and 5A and 6A group element which are used for a raw material, a nitride, As powder of boride, TiC, ZrC, HfC, VC, NbC, TaC, Cr₃C₂, Mo₂C, WC, TiN, ZrN, HfN, VN, NbN, Each powder of TaN, Cr₃N₂, Mo₂N, WN, TiB₂, ZrB₂, HfB₂, VB₂, NbB₂ and TaB₂, Cr₃ B₄, MoB₂, and WB₂ grade, Or among these, the mixed powder which consists of two or more sorts, or TiC, ZrC, HfC, VC, NbC, TaC, Cr₃C₂, Mo₂C, WC, TiN, ZrN, HfN, VN, NbN, TaN, Cr₃N₂, Mo₂N, WN, TiB₂, ZrB₂, HfB₂, VB₂, NbB₂ and TaB₂, Cr₃ B₄, and the solid-solution powder that consists of any two or more sorts, MoB₂ and WB₂, are used. the loadings in the inside of the raw material mixture of any one or more sorts of powder of the carbide of this Si and the periodic tables 4A and 5A, and 6A group element, a nitride, and boride -- ten to 30 capacity % -- it considers as 15 to 25 capacity % more desirably. Since the content ratio of a difficulty sintering component will serve as size and eburnation will stop being able to progress quite easily if the composite material with which the elevated-temperature property common to said component, i.e., an elevated-temperature quantity degree of hardness, elevated-temperature high intensity, etc. were reflected in general enough becomes difficult to get and 30 capacity % is exceeded, it is not desirable at under 10 capacity %.

[0015] As a metal aluminium powder, the high grade powder of commercial metal aluminum can be used, and the loadings in the raw material mixture are made into 0.1 to 10 capacity %. Since Melting aluminum may deposit in a base material phase and the mechanical property of composite material may deteriorate remarkably when the loadings of a metal aluminium powder serve as a fall of the consistency of a sintered compact, or reinforcement at less than 0.1 % of the weight and 10 capacity % is exceeded, it is not desirable.

[0016] moreover, as a compound containing the oxygen and silicon of Li, Na, and K which are used for a raw material For example, Li₂SiO₃, Li₄SiO₄, Li₂SiO₃andH₂O, Li₆Si₂O₇, Na₂SiO₃, Na₄SiO₄, Na₂Si₂O₅, Na₂Si₄O₉, Na₂O-xSiO₂ and nH₂O (however, x=2-4, n>=1), and K₂SiO₃ grade -- it can mention -- among these -- since -- what was chosen suitably is used. The loadings in the raw material mixture are made into 0.1 to 8 capacity %. Since eburnation runs short, the integrated state of a cBN grain and a base material phase falls, for example, it becomes easy to produce a fall on the strength, degraining, etc., it is not desirable at under 0.1 capacity %. Moreover, since the rate of the low-melt point point matter containing any one or more sorts of Li, Na, and K which exist in a sintered compact may increase, a degree of hardness may be begun and mechanical properties, such as reinforcement and toughness, may deteriorate when 8 capacity % is exceeded, it is not desirable. In addition, the alkali metal of said outside, i.e., the compound

which contains the oxygen and silicon also about Rb and Cs, can be used similarly.

[0017] The detail of a production process until it obtains cubic boron nitride content hard composite material using these raw materials is shown below. each raw material component becomes said predetermined loadings -- as -- weighing capacity -- it mixes and raw material mixture is produced. Although mixing of a raw material can respond by mixing in a ball mill using organic solvents, such as well-known wet blending, for example, ethyl alcohol, or isopropyl alcohol, it can also use inorganic system solvents, such as water, instead of an organic solvent in this invention. Subsequently, this raw material mixture is suitably dried by reduced pressure drying, spray drying, etc. if needed, and pressure sintering of the moldings which fabricated desiccation raw material mixture by press forming etc. in the desired configuration is carried out, or pressure sintering of the non-fabricated raw material mixture is put in and carried out to the cel for pressurization of a request configuration.

[0018] Pressure sintering is performed at the pressure of 100 or more MPas, and the temperature of 1000-1500 degrees C. For example, although a pressure 5000 - 6000MPa, and the temperature of 1400-1500 degrees C will serve as general conditions if cBN carries out pressure sintering in the field which exists in stability independently Remarkable reduction-ization of a manufacturing cost can be attained in the comparatively low pressure considered to exist metastable if cBN is independent, and a temperature field. In this invention, especially the thing done for pressure sintering of it on the pressure and temperature conditions which are considered to exist metastable if cBN is independent since it can suppress enough carrying out phase transition also in such pressurization temperature conditions to hBN whose cBN is a brittle phase in composite material is recommended. It is good to carry out at a pressure 500 - 2000MPa, and the temperature of 1100-1250 degrees C preferably as such conditions. Since it is easy to become that sintering progresses at the temperature of less than 1000 degrees C insufficiently being sufficiently hard of eburnation on the other hand, the case where temperature exceeds 1500 degrees C conversely, and a pressure serve as a field where cBN is made into astable in less than 100 MPas, and the phase transition to hBN which is a brittle phase may arise, it is not desirable. Moreover, although it depends for the holding time of pressure sintering on a pressure and temperature, it can respond in about 5 - 30 minutes in general. In addition, although it is not limited especially unless it is the high oxidizing atmosphere which causes oxidation of the non-oxide component of cBN or a base material phase directly about the ambient atmosphere at the time of sintering, for example, it is possible also in air, it is good to sinter in inert gas, such as inside of a vacuum, and argon gas, more desirably.

[0019] Which equipment may be used as long as it is equipment which can generate the pressure of 100 or more MPas, and the temperature of 1000 degrees C or more as equipment used for such pressure sintering. As an example of such equipment, the high pressure of hotpress equipment, HIP equipment, a piston cylinder mold, a flat belt mold, a girdle mold, the Bridgman mold, a cubic Annville mold, etc. or extra-high voltage heating apparatus can be mentioned. The cBN particle which does not contain hBN by performing pressure sintering with such conditions and equipment An aluminum oxide, the mullite of a minute amount and its solid solution, Si, and periodic table 4A, Any one or more sorts of components of the carbide of 5A and 6A group element, a nitride, and boride are made into a subject. the quantity distributed in the continuous base material which consists of what any one or more sorts of the matter which contains in this the oxygen and/or silicon of Li, Na, and K which are alkali metal joined -- a precise cubic boron nitride content hard composite material can be manufactured.

[0020]

[An operation and effectiveness] The compound containing the oxygen and silicon of alkali metal like Li, Na, and K in this invention Set the front stirrup of pressure sintering to an initial stage, and it reacts or dissolves with a cBN particle and other base material phase constituents, respectively. Increase the labile between a cBN particle and a base material phase, consequently it is thought that a comparatively firm and chemical integrated state is formed between a dispersed phase and a base material phase in a compound sintered compact. As a result, a cBN particle is strongly held to a base material phase in a sintered compact, and degraining stops being able to happen easily, and also it leads to the improvement in on the strength of a sintered compact. Furthermore, in a **** process, some silicic acid components of the compound containing the oxygen and silicon of alkali metal dissociate, this silicic acid component reacts especially with the aluminum-oxide surface section in a base material phase, and the mullite which is a high-melting compound,

and its solid solution are generated to the grain boundary of an aluminum oxide. This mullite has a low coefficient of thermal expansion compared with other high-melting hard mineral matter which constitutes a base material phase, and raises the thermal shock resistance of a base material phase.

[0021] Moreover, since the dispersion effect in the inside of a drainage system solvent is excellent, by this invention, the water which is, the solvents, i.e., the inorganic system solvent, other than an organic system, can also be used for the silicate of alkali metal in a raw material adjustment phase. Therefore, handling becomes simpler and is connected also with reduction-ization of cost, and also when an organic system solvent is used, sintering somatic degradation by the residual carbon impurity after often produced heating can be avoided.

[0022] Furthermore, according to this invention, even if it does not cover cBN powder for example, with a high-melting inorganic compound beforehand, it can manufacture the sufficiently precise compound sintered compact of a high degree of hardness, without generating hBN at the pressure and temperature which are called metastable field of cBN. The compound which contains the alkali metal and silicon of a preceding paragraph story to a low-melt point point of low-temperature sintering comparatively near the interface of a cBN grain about this reason reacts with the surface section of a cBN body, and it is thought by forming this resultant in a cBN front face that the phase transition to hBN is controlled. thus -- especially this method is [equipment / expensive / that a sintering pressure can be made lower than the conventional approach] unnecessary in a **** covering process -- carrying out -- moreover -- comparatively -- easy -- quantity -- since a precise sintered compact can be manufactured, large reduction-ization of the large improvement in productivity and a production cost is attained. the quantity which this invention equipped not only with a high degree of hardness and high abrasion resistance but with the outstanding toughness, defect resistance, reinforcement, and workability -- since a precise composite material can be obtained, it is the manufacture approach for which it was extremely suitable also as an object for member manufacture of an antifriction application metallurgy group cutting application.

[0023]

[Example] Hereafter, an example and the example of a comparison explain this invention concrete more in detail.

[Example 1] Aluminum oxide dust with a mean particle diameter of 0.5 micrometers, a metal aluminium powder with a mean particle diameter of 3.0 micrometers, The titanium nitride powder (99.5% of purity) of 1 micrometer of mean diameters, the boronizing tantalum powder of 1 micrometer of mean diameters (99.5% of purity), Tungsten carbide powder (99.5% of purity) with a mean particle diameter of about 1 micrometer, silicon nitride powder with a mean particle diameter of about 1 micrometer (99.5% of purity), The cBN powder of about 3 micrometers of mean diameters, the meta-silicic acid lithium powder of about 5 micrometers of mean diameters, Specific metasilicate powder with a mean particle diameter of about 10 micrometers, the specific metasilicate hydrate of a mucoid, It puts into the pot made from an alumina, respectively, and water is used with alumina balls for 72 hours so that it may become seven kinds of raw material blending ratio of coal which described meta-potassium silicate powder with a mean particle diameter of about 5 micrometers in Table 1. After wet blending, Spray drying of the mixture was carried out, granulation was created, and this was fabricated in press forming with a diameter thickness [about 2cm thickness of about 5cm] in the shape of a cylindrical shape. Pressure sintering of this moldings was carried out for 15 minutes at 1000MPa and 1250 degrees C among air using the piston cylinder mold high-pressure sintering furnace. Although powder X-ray **** analyzed the component of the obtained sintered compact (this invention article 1-7), the mullite of the component which hBN is not detected but makes a subject the high-melting matter and cBN corresponding to each raw material presentation for all in general, and a minute amount was detected.

[0024]

[Table 1]

本発明品	原料配合量		焼結体機械的性質
	原料名	容量%	
1	cBN	37.7	相対密度 99.9% ビッカース硬度 2900 曲げ強度 145MPa
	Al ₂ O ₃	32.5	
	Al	3.2	
	TiN	25.2	
	Li ₂ SiO ₃	1.4	
2	cBN	52.7	相対密度 99.9% ビッカース硬度 3000 曲げ強度 125MPa
	Al ₂ O ₃	21.8	
	Al	2.0	
	TiN	15.5	
	Na ₂ SiO ₃	8.0	
3	cBN	45.4	相対密度 99.9% ビッカース硬度 2800 曲げ強度 145MPa
	Al ₂ O ₃	22.4	
	Al	2.1	
	TiN	30.0	
	K ₂ SiO ₃	0.1	
4	cBN	30.0	相対密度 99.9% ビッカース硬度 2650 曲げ強度 130MPa
	Al ₂ O ₃	50.0	
	Al	3.2	
	TaB ₅	15.3	
	Na ₂ SiO ₃ ・2H ₂ O	1.5	
5	cBN	50.0	相対密度 99.9% ビッカース硬度 2950 曲げ強度 120MPa
	Al ₂ O ₃	20.0	
	Al	10.0	
	TiN	19.0	
	Na ₂ SiO ₃	1.0	
6	cBN	46.3	相対密度 99.9% ビッカース硬度 2750 曲げ強度 125MPa
	Al ₂ O ₃	41.6	
	Al	0.1	
	WC	11.9	
	Na ₂ SiO ₃	0.1	
7	cBN	65.0	相対密度 99.9% ビッカース硬度 2950 曲げ強度 140MPa
	Al ₂ O ₃	20.0	
	Al	2.5	
	Si ₃ N ₄	10.0	
	Na ₂ SiO ₃	2.5	

[0025] Moreover, the mechanical property of this sintered compact is measured as the following, and measured value is described in Table 1. In addition, it computed from the relative bulk density and true specific gravity which were measured by the approach which applied to JIS-C2141 correspondingly about relative density, the indenter load was measured as 5kg about Vickers hardness, and the tripartite flexural strength (room temperature) based on JIS-R1601 was measured about flexural strength.

[0026] [Example 2] Aluminum-oxide-dust 26 with a mean particle diameter of 0.5 micrometers capacity %, metal aluminium-powder 2.5 with a mean particle diameter of 3.0 micrometers capacity %, titanium nitride powder (99.5% of purity) 20.1 with a mean particle diameter of about 1 micrometer capacity %, specific metasilicate powder 1.1 with a mean particle diameter of about 8 micrometers capacity %, and with a mean particle diameter of about 3 micrometers cBN powder 50.3 capacity % were used as the raw material, and the non-calcinated moldings of the shape of a with a diameter thickness [about 2cm thickness of about 5cm] cylindrical shape be produced by the same approach as said example 1 Pressure sintering was performed on the conditions described in Table 2 with the isostatic pressing equipment (HIP) between heat which carried out pressure sintering on the conditions which describe this in Table 2 with a piston cylinder mold high-pressure sintering furnace, or (this invention article 8-13) carried out degassing enclosure of this moldings into the container made from SUS310, and used Ar gas as the pressure medium (this invention article 14), and the sintered compact was produced.

[0027]

[Table 2]

本発明品	加圧焼結条件			焼結体機械的性質
	温度 (°C)	圧力 (MPa)	焼結装置	
8	1000	1000	ピストンシリンダー型圧焼結炉	相対密度 99.9% ビッカース硬度 2750 曲げ強度 135MPa
9	1100	1000	ピストンシリンダー型圧焼結炉	相対密度 99.9% ビッカース硬度 2750 曲げ強度 136MPa
10	1200	1000	ピストンシリンダー型圧焼結炉	相対密度 99.9% ビッカース硬度 3050 曲げ強度 140MPa
11	1500	1000	ピストンシリンダー型圧焼結炉	相対密度 99.9% ビッカース硬度 3000 曲げ強度 135MPa
12	1250	2000	ピストンシリンダー型圧焼結炉	相対密度 99.9% ビッカース硬度 3100 曲げ強度 140MPa
13	1250	500	ピストンシリンダー型圧焼結炉	相対密度 99.9% ビッカース硬度 2900 曲げ強度 130MPa
14	1250	100	HIP	相対密度 99.7% ビッカース硬度 2700 曲げ強度 125MPa

[0028] although powder X-ray **** analyzed the component of the obtained sintered compact (this invention article 8-14), hBN detects all -- not having -- cBN and aluminum2 -- the mullite of the component which makes O3 and TiN a subject, and a minute amount was detected. Moreover, the result of having measured the mechanical property of this sintered compact by the same approach as an example 1 is described in Table 2.

[0029] [Example 1 of a comparison] Aluminum-oxide-dust 33.9 with a mean particle diameter of 0.5 micrometers capacity %, metal aluminium-powder 3.2 with a mean particle diameter of 3.0 micrometers capacity %, titanium nitride powder (99.5% of purity) 25.2 with a mean particle diameter of about 1 micrometer capacity %, and with a mean particle diameter of about 3 micrometers cBN powder 37.7 capacity % were used as the raw material, and the sintered compact of the shape of a with a diameter thickness [about 2cm thickness of about 5cm] cylindrical shape was produced on condition that the same approach as an example 1, and pressure sintering. As a result of measuring the mechanical characteristics of this sintered compact by the same approach as an example 1, it was set to 99.3% of relative density, Vickers hardness 2750, and flexural strength 100MPa, and flexural strength became quite low.

[0030] [Example 2 of a comparison] Aluminum-oxide-dust 33.9 with a mean particle diameter of 0.5 micrometers capacity %, metal aluminium-powder 3.2 with a mean particle diameter of 3.0 micrometers capacity %, titanium nitride powder (99.5% of purity) 25.2 with a mean particle diameter of about 1 micrometer capacity %, and with a mean particle diameter of about 3 micrometers cBN powder 37.7 capacity % were used as the raw material, and the non-calcinated moldings of the shape of a with a diameter thickness [about 2cm thickness of about 5cm] cylindrical shape was produced by the same approach as said example 1. Pressure sintering was carried out at pressure 100MPa and the temperature of 1250 degrees C for 60 minutes with the isostatic pressing equipment (HIP) between heat which made this degassing enclosure into the container made from SUS310, and used Ar gas as the pressure medium. mechanical [set to 92% of relative density, Vickers hardness 2300, and flexural strength 105MPa and], as a result of powder X-ray *****s analyzing the component of the obtained sintered compact, and detecting hBN and measuring the mechanical characteristics of this sintered compact by the same approach as an example 1 -- description became a remarkable low thing.

[Translation done.]